

CLAIMS

1. A microencapsulated catalyst-ligand system comprising a catalyst and a ligand microencapsulated within a permeable polymer microcapsule shell.
2. A microencapsulated catalyst-ligand system comprising a catalyst and a ligand microencapsulated within a permeable polymer microcapsule shell wherein the microcapsule shell is formed by interfacial polymerisation.
3. A microencapsulated catalyst-ligand system obtainable by a process comprising forming a permeable microcapsule shell by interfacial polymerisation in the presence of a catalyst and a ligand.
4. A microencapsulated catalyst-ligand system according to any one of Claims 1 to 3 wherein the permeable polymer microcapsule shell is the product of self-condensation and/or cross-linking of etherified urea-formaldehyde resins or prepolymers in which from about 50 to about 98% of the methylol groups have been etherified with a C<sub>4</sub>-C<sub>10</sub> alcohol.
5. A microencapsulated catalyst-ligand system according to any one of Claims 1 to 3 wherein the permeable polymer microcapsule shell is a polyurea microcapsule prepared from at least one polyisocyanate and/or tolylene diisocyanate.
6. A microencapsulated catalyst-ligand system according to Claim 5 wherein the polyisocyanates and/or tolylene diisocyanates are selected from the group consisting of 1-chloro-2,4-phenylene diisocyanate, m-phenylene diisocyanate (and its hydrogenated derivative), p-phenylene diisocyanate (and its hydrogenated derivative), 4,4'-methylenebis(phenyl isocyanate), 2,4-tolylene diisocyanate, tolylene diisocyanate (60% 2,4-isomer, 40% 2,6-isomer), 2,6-tolylene diisocyanate, 3,3'-dimethyl-4,4'-biphenylene diisocyanate, 4,4'-methylenebis (2-methylphenyl isocyanate), 3,3'-dimethoxy-4,4'-biphenylene diisocyanate, 2,2',5,5'-tetramethyl-4,4'-biphenylene diisocyanate, 80% 2,4- and 20% 2,6-isomer of tolylene diisocyanate, polymethylene polyphenylisocyanate (PMPPI), 1,6-hexamethylene diisocyanate, isophorone diisocyanate, tetramethylxylene diisocyanate and 1,5-naphthylene diisocyanate.
7. A microencapsulated catalyst-ligand system according to any one of Claims 1 to 6 wherein the catalyst is an inorganic catalyst, preferably a transition metal catalyst.
8. A microencapsulated catalyst-ligand system according to Claim 7 wherein the catalyst is a transition metal catalyst wherein the transition metal is platinum, palladium,

osmium, ruthenium, rhodium, iridium, rhenium, scandium, cerium, samarium, yttrium, ytterbium, lutetium, cobalt, titanium, chromium, copper, iron, nickel, manganese, tin, mercury, silver, gold, zinc, vanadium, tungsten and molybdenum.

5 9. A microencapsulated catalyst-ligand system according to Claim 8 wherein the catalyst is a transition metal catalyst wherein the transition metal is palladium, preferably the palladium is in the form of an organic solvent soluble form and most preferably is palladium acetate.

10 10. A microencapsulated catalyst-ligand system according to any one of Claims 1 to 9 wherein the ligand is an organic moiety comprising one or more hetroatoms selected from N, O, P and S.

15 11. A microencapsulated catalyst-ligand system according to Claim 10 wherein the ligand is an organic ligand of formula (1):

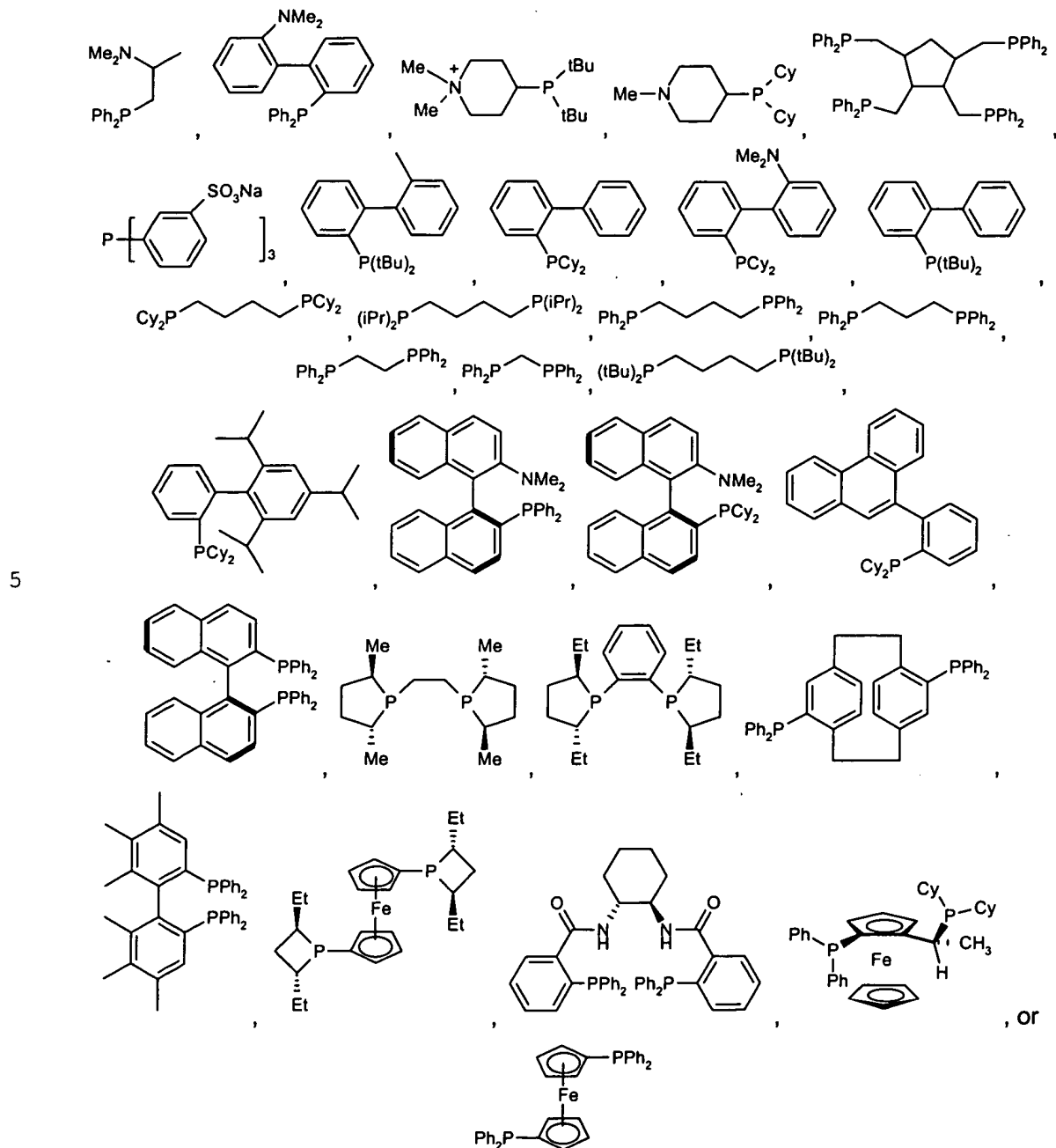


(1)

wherein:

20  $R^1$ ,  $R^2$  and  $R^3$  are each independently an optionally substituted hydrocarbyl group, an optionally substituted hydrocarbyloxy group, or an optionally substituted hetrocyclyl group or one or more of  $R^1$  &  $R^2$ ,  $R^1$  &  $R^3$ ,  $R^2$  &  $R^3$  optionally being linked in such a way as to form an optionally substituted ring(s).

25 12. A microencapsulated catalyst-ligand system according to Claim 11 wherein the ligand is  $PMe_2CF_3$ ,  $P(OEt)_3$ ,  $P(Et)_3$ ,  $P(Bu)_3$ ,  $P(cyclohexyl)_3$ ,  $PPhEt_2$ ,  $PPh_2Me$ ,  $PPh_3$ ,  $P(CH_2Ph)_3$ ,  $P(CH_2Ph)Ph_2$ ,  $P(p\text{-tolyl})_3$ ,  $P(o\text{-}C_6H_4OMe)_3$ ,  $P(OPh)_3$ ,  $P(O\text{-}p\text{-tolyl})_3$ ,  $P(p\text{-}C_6H_4OMe)_3$ ,  $P(o\text{-tolyl})_3$ ,  $P(m\text{-tolyl})_3$ ,  $PMe_3$ ,  $PPhMe_2$ ,  $PPh_2Et$ ,  $P(i\text{-}Pr)_3$ ,  $P(t\text{-}Bu)_3$ ,  $PPhCH_2Ph$ ,  $PPh_2OEt$ ,  $PPh(OEt)_2$ ,  $P(O\text{-}o\text{-tolyl})_3$ ,  $P(OMe)_3$ ,  $P(n\text{-}Pr)_3$ ,  $PPh(i\text{-}Pr)_2$ ,  $PPh_2(i\text{-}Pr)$ ,  
30  $PPhBu_2$ ,  $PPh_2Bu$ ,  $P(i\text{-}Bu)_3$ ,  $PPh(cyclohexyl)_2$ ,  $PPh_2(cyclohexyl)$ ,  $P(CH_2Ph)_2Et$ ,  $P(CH_2Ph)Et_2$ ,  $P(C_6F_5)Ph_2$ ,  $P(p\text{-}C_6H_4F)_3$ ,  $P(p\text{-}C_6H_4Cl)_3$ ,  $P(C_6F_5)_2Ph$ ,  $P(o\text{-}C_6H_4F)_3$ ,  $P(o\text{-}C_6H_4Cl)_3$ ,  $P(2\text{-furyl})_3$ ,  $P(2\text{-thienyl})_3$ ,  $P(n\text{-octyl})_3$ ,  $P(p\text{-}C_6H_4NO_2)_3$ ,



where Cy = cyclohexyl.

10

13. A process for the preparation of a microencapsulated catalyst-ligand system which comprises forming a microcapsule shell by interfacial polymerisation in the presence of a catalyst and a ligand.

15

14. A process for the preparation of a microencapsulated catalyst-ligand system which comprises

- (a) dissolving or dispersing the catalyst and ligand in a first phase,
  - (b) dispersing the first phase in a second, continuous phase to form an emulsion,
  - (c) reacting one or more microcapsule wall-forming materials at the interface between the dispersed first phase and the continuous second phase to form a microcapsule polymer shell encapsulating the dispersed first phase core and optionally
  - (d) recovering the microcapsules from the continuous phase.
- 10 15. A microencapsulated catalyst-ligand system obtainable by a process comprising forming a microcapsule shell by interfacial polymerisation in the presence of a catalyst and treating the microcapsule shell with a ligand.
- 15 16. A process for the preparation of a microencapsulated catalyst-ligand system which comprises
- (a) dissolving or dispersing the catalyst in a first phase,
  - (b) dispersing the first phase in a second, continuous phase to form an emulsion,
  - (c) reacting one or more microcapsule wall-forming materials at the interface between the dispersed first phase and the continuous second phase to form a microcapsule polymer shell encapsulating the dispersed first phase core; and
  - (d) treating the microcapsules with a ligand.
- 20
- 25 17. A microencapsulated catalyst-ligand system obtainable by a process comprising forming a microcapsule shell by interfacial polymerisation in the presence of a ligand and treating the microcapsule shell with a catalyst solution.
- 30 18. A process for the preparation of a microencapsulated catalyst-ligand system which comprises
- (a) dissolving or dispersing the ligand in a first phase,
  - (b) dispersing the first phase in a second, continuous phase to form an emulsion,
  - (c) reacting one or more microcapsule wall-forming materials at the interface between the dispersed first phase and the continuous second phase to form a microcapsule polymer shell encapsulating the dispersed first phase core; and
  - (d) treating the microcapsules with a solution of a catalyst.
- 35

19. A process according to any one of Claims 13, 15 or 17 wherein the interfacial polymerisation comprises self-condensation and/or cross-linking of etherified urea-formaldehyde resins or prepolymers in which from about 50 to about 98% of the methylol groups have been etherified with a C<sub>4</sub>-C<sub>10</sub> alcohol.

20. A process according to any one of Claims 13, 15 or 17 wherein the interfacial polymerisation comprises condensation of at least one polyisocyanate and/or tolylene diisocyanate.

21. A process according to Claim 20 wherein the polyisocyanates and/or tolylene diisocyanates are selected from the group consisting of 1-chloro-2,4-phenylene diisocyanate, m-phenylene diisocyanate (and its hydrogenated derivative), p-phenylene diisocyanate (and its hydrogenated derivative), 4,4'-methylenebis(phenyl isocyanate), 2,4-tolyene diisocyanate, tolylene diisocyanate (60% 2,4-isomer, 40% 2,6-isomer), 2,6-tolyene diisocyanate, 3,3'-dimethyl-4,4'-biphenylene diisocyanate, 4,4'-methylenebis (2-methylphenyl isocyanate), 3,3'-dimethoxy-4,4'-biphenylene diisocyanate, 2,2',5,5'-tetramethyl-4,4'-biphenylene diisocyanate, 80% 2,4- and 20% 2,6-isomer of tolylene diisocyanate, polymethylene polyphenylisocyanate (PMPPi), 1,6-hexamethylene diisocyanate, isophorone diisocyanate, tetramethylxylene diisocyanate and 1,5-naphthylene diisocyanate.

22. A process according to Claim 20 or Claim 21 wherein a crosslinking reagent is present.

23. A process according to any one of Claims 19 to 22 wherein unreacted amine groups are converted to urea, amide or urethane groups by post reaction with a monoisocyanate, acid chloride or chloroformate.

24. A process according to any one of Claims 14, 16 or 18 wherein the wall-forming materials comprise etherified urea-formaldehyde resins or prepolymers in which from about 50 to about 98% of the methylol groups have been etherified with a C<sub>4</sub>-C<sub>10</sub> alcohol.

25. A process according to any one of Claims 14, 16 or 18 wherein the wall-forming materials comprise at least one polyisocyanate and/or tolylene diisocyanate.

26. A process according to Claim 25 wherein the polyisocyanates and/or tolylene diisocyanates are selected from the group consisting of 1-chloro-2,4-phenylene diisocyanate, m-phenylene diisocyanate (and its hydrogenated derivative), p-phenylene diisocyanate (and its hydrogenated derivative), 4,4'-methylenebis(phenyl isocyanate), 2,4-

tolylene diisocyanate, tolylene diisocyanate (60% 2,4-isomer, 40% 2,6-isomer), 2,6-tolylene diisocyanate, 3,3'-dimethyl-4,4'-biphenylene diisocyanate, 4,4'-methylenebis (2-methylphenyl isocyanate), 3,3'-dimethoxy-4,4'-biphenylene diisocyanate, 2,2',5,5'-tetramethyl-4,4'-biphenylene diisocyanate, 80% 2,4- and 20% 2,6-isomer of tolylene diisocyanate, polymethylene polyphenylisocyanate (PMPPI), 1,6-hexamethylene diisocyanate, isophorone diisocyanate, tetramethylxylene diisocyanate and 1,5-naphthylene diisocyanate.

27. A process according to Claim 25 or Claim 26 wherein the wall-forming materials comprise a crosslinking reagent.

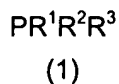
28. A process according to any one of Claims 13 to 27 wherein the catalyst is an inorganic catalyst, preferably a transition metal catalyst.

29. A process according to Claim 28 wherein the catalyst is a transition metal catalyst wherein the transition metal is platinum, palladium, osmium, ruthenium, rhodium, iridium, rhenium, scandium, cerium, samarium, yttrium, ytterbium, lutetium, cobalt, titanium, chromium, copper, iron, nickel, manganese, tin, mercury, silver, gold, zinc, vanadium, tungsten and molybdenum.

30. A process according to Claim 29 wherein the catalyst is a transition metal catalyst wherein the transition metal is palladium, preferably the palladium is in the form of an organic solvent soluble form and most preferably is palladium acetate.

31. A process according to any one of Claims 13 to 30 wherein the ligand is an organic moiety comprising one or more heteroatoms selected from N, O, P and S.

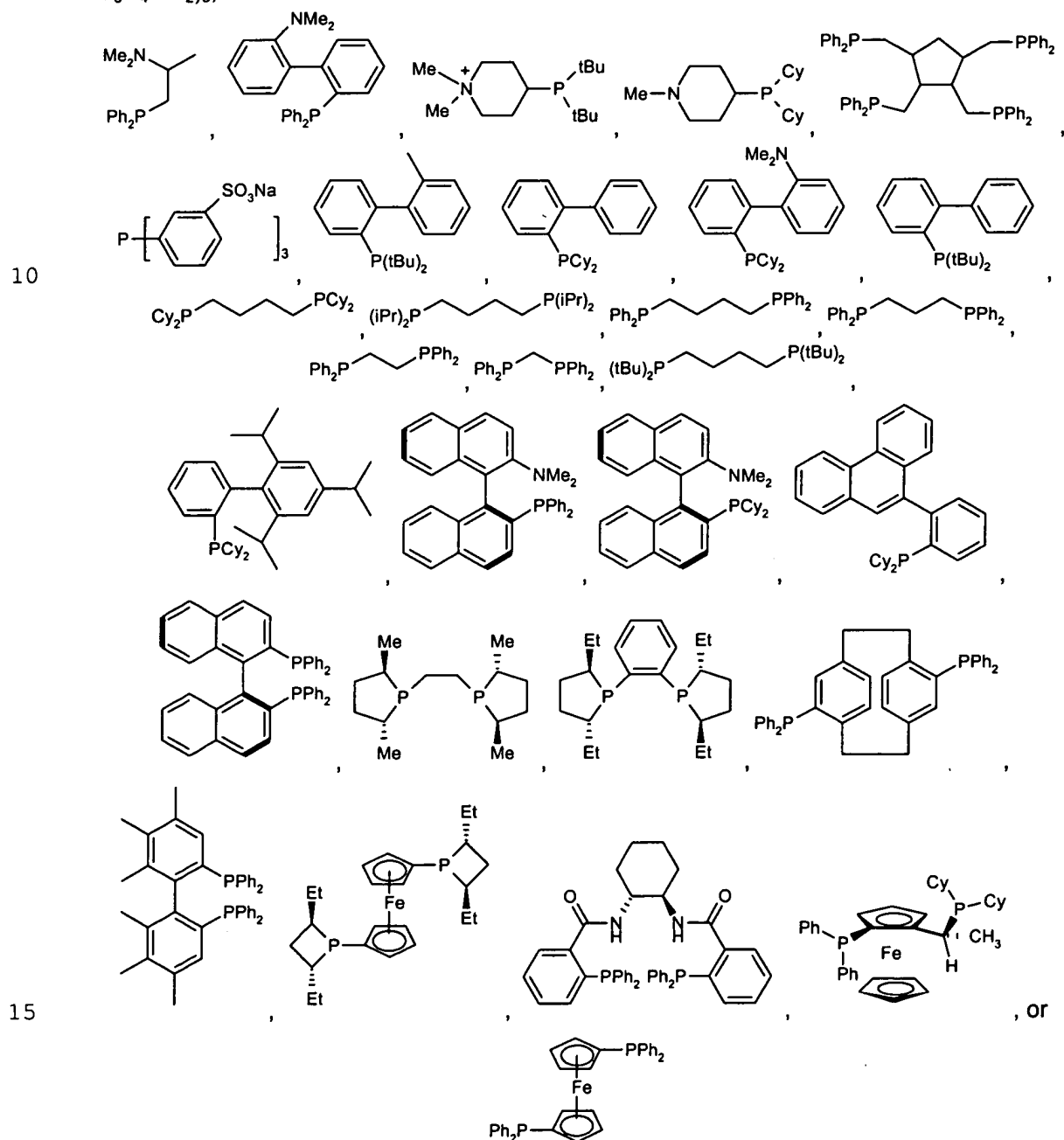
32. A process according to Claim 31 wherein the ligand is an organic ligand of formula (1):



wherein:

$\text{R}^1$ ,  $\text{R}^2$  and  $\text{R}^3$  are each independently an optionally substituted hydrocarbyl group, an optionally substituted hydrocarbyloxy group, or an optionally substituted heterocyclyl group or one or more of  $\text{R}^1$  &  $\text{R}^2$ ,  $\text{R}^1$  &  $\text{R}^3$ ,  $\text{R}^2$  &  $\text{R}^3$  optionally being linked in such a way as to form an optionally substituted ring(s).

33. A process according to Claim 32 wherein the ligand is  $\text{PMe}_2\text{CF}_3$ ,  $\text{P}(\text{OEt})_3$ ,  $\text{P}(\text{Et})_3$ ,  $\text{P}(\text{Bu})_3$ ,  $\text{P}(\text{cyclohexyl})_3$ ,  $\text{PPhEt}_2$ ,  $\text{PPh}_2\text{Me}$ ,  $\text{PPh}_3$ ,  $\text{P}(\text{CH}_2\text{Ph})_3$ ,  $\text{P}(\text{CH}_2\text{Ph})\text{Ph}_2$ ,  $\text{P}(p\text{-tolyl})_3$ ,  $\text{P}(o\text{-C}_6\text{H}_4\text{OMe})_3$ ,  $\text{P}(\text{OPh})_3$ ,  $\text{P}(\text{O-}p\text{-tolyl})_3$ ,  $\text{P}(p\text{-C}_6\text{H}_4\text{OMe})_3$ ,  $\text{P}(o\text{-tolyl})_3$ ,  $\text{P}(m\text{-tolyl})_3$ ,  $\text{PMe}_3$ ,  $\text{PPhMe}_2$ ,  $\text{PPh}_2\text{Et}$ ,  $\text{P}(i\text{-Pr})_3$ ,  $\text{P}(t\text{-Bu})_3$ ,  $\text{PPhCH}_2\text{Ph}$ ,  $\text{PPh}_2\text{OEt}$ ,  $\text{PPh}(\text{OEt})_2$ ,  $\text{P}(\text{O-}o\text{-tolyl})_3$ ,  $\text{P}(\text{OMe})_3$ ,  $\text{P}(n\text{-Pr})_3$ ,  $\text{PPh}(i\text{-Pr})_2$ ,  $\text{PPh}_2(i\text{-Pr})$ ,  $\text{PPhBu}_2$ ,  $\text{PPh}_2\text{Bu}$ ,  $\text{P}(i\text{-Bu})_3$ ,  $\text{PPh}(\text{cyclohexyl})_2$ ,  $\text{PPh}_2(\text{cyclohexyl})$ ,  $\text{P}(\text{CH}_2\text{Ph})_2\text{Et}$ ,  $\text{P}(\text{CH}_2\text{Ph})\text{Et}_2$ ,  $\text{P}(\text{C}_6\text{F}_5)\text{Ph}_2$ ,  $\text{P}(p\text{-C}_6\text{H}_4\text{F})_3$ ,  $\text{P}(p\text{-C}_6\text{H}_4\text{Cl})_3$ ,  $\text{P}(\text{C}_6\text{F}_5)_2\text{Ph}$ ,  $\text{P}(o\text{-C}_6\text{H}_4\text{F})_3$ ,  $\text{P}(o\text{-C}_6\text{H}_4\text{Cl})_3$ ,  $\text{P}(2\text{-furanyl})_3$ ,  $\text{P}(2\text{-thienyl})_3$ ,  $\text{P}(n\text{-octyl})_3$ ,  $\text{P}(p\text{-C}_6\text{H}_4\text{NO}_2)_3$ .



where Cy = cyclohexyl.